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(54) BENDIOCARB GRANULES

(72) Lemon, Roger W.,
U.K.

(73) Granted to Fisons Limited
U.K.

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Case 2953

This invention relates to combating pests.

The invention provides a method of combating pests at a locus at which a brassica crop is growing or is to grow, which method comprises applying to the locus a pest-combating amount of granules containing bendiocarb.

The invention also provides a mixture of brassica seed and granules containing bendiocarb, particularly where the seed and granules are of similar size and density. The invention also provides per se these granules of similar size and density, together with their production.

Bendiocarb is the known pesticide 2,2-dimethyl-1,3-benzodioxol-4-yl methylcarbamate. It has been discovered that by employing it in the form of granules, it is remarkably useful against brassica pests. In this use, the granules exhibit a surprisingly good combination of high activity against the crop pests and high safety to the crop.

The crop may be, for instance, Brussels sprouts, cabbages, cauliflowers, mustard, kale, kohl rabi, broccoli, radishes, turnips or swede. Preferably, however, the brassica is rape, e.g. Brassica napus. The rape may be fodder rape, or, preferably, oil seed rape.

The pest animals are usually arthropods, particularly

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insects or acarids, especially insects. Particularly important is combating brassica flea beetle (e.g. Phyllotreta cruciferae) or cabbage stem beetle (e.g. Psylliodes chrysocephala). Cabbage root fly (Erioischia brassicae), stem weevil (Ceutorhynchus quadridens) or gall weevil (Ceutorhynchus pleurostigma) are also combated, as are soil pests such as wireworms, symphylids or leatherjackets.

Granules are used in the present invention. These are of larger particle size than powders, e.g. wettable powders. Preferably no more than 4% of the granules is smaller than 250 microns in major dimension.

The granules can be applied before or after sowing the brassica seed. Preferably, however, the granules are applied while sowing the seed. The granules can be applied to the seedbed by broadcasting the granules or by using a granule applicator. Preferably, however, the granules and seed are mixed and sown together. Usually 50-1000g, preferably 100-500g, of bendiocarb are applied per hectare.

Usually the present mixtures of granules and seed, especially rape seed, are in the proportion of granules: seed of 1:10 to 10:1, preferably 1:4 to 2:1, by weight.

The granules usually contain 1-15%, preferably 3-8%, e.g. 5%, of bendiocarb. Percentages in this specification

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are by weight unless otherwise indicated.

The granules may contain an additional pesticide to the bendiocarb, to act on the locus being treated, e.g. another insecticide or acaricide, particularly another insecticide, but this is not preferred.

The granules can be of conventional type.

The granules can comprise absorbent base granules impregnated with the bendiocarb. Preferably, however, the bendiocarb granules comprise base granules to which the bendiocarb adheres by means of a sticker.

It is especially preferred that the granules be of similar size and bulk density to the brassica seed, so that the two can be applied to the soil as a mixture through the same application equipment without the two separating from each other to cause uneven application. It can readily be tested whether given granules and seed are such similar bulk density by mixing them to form a uniform mixture and then shaking to see if the mixture becomes non-uniform. These granules, and the mixture of granule and seed are novel, and the invention provides them per se. The bulk density of the granules is generally 0.4 to 0.8 preferably 0.40 to 0.65 g/ml as measured according to CIPAC test method MT 58.4. The granules are generally substantially all of size 400 - 2500, preferably 600 - 2500, especially 1000 - 2000, microns in major dimension. Sieve sizes as referred to

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i- this specification are according to British Standard 410.

In a preferred embodiment, the invention provides granules containing bendiocarb which are of bulk density 0.45 to 0.60 g/ml and which are substantially all of size 1000-2000 microns in major dimension. These are particularly suitable for admixture with rape seed.

The novel granules are preferably of mean major dimension (ie the mean on a weight basis of the major dimension of each granule) 1000-2000 microns.

In a preferred embodiment, the invention provides granules which are of bulk density 0.45 to 0.60 g/ml and which are of mean major dimension 1000-2000 microns. These are particularly suitable for admixture with rape seed.

The novel granules can comprise absorbent base granules impregnated with the bendiocarb, or base granules to which the bendiocarb adheres by means of a sticker. The latter are preferred. In either type, the base granules are preferably botanical base granules, e.g. walnut shell, peanut shell, wood chips or especially corn cob grit. The granules usually contain 75-99% of base granules. When botanical base granules are employed, the present granules may be susceptible in some circumstances to attack by fungus. Accordingly, the

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present granules containing botanical base granules may contain a fungicide, e.g. methyl p-hydroxybenzoate or a Dowicide* (from Dow Chemical) to protect them from such attack.

The novel impregnated granules can be prepared by dissolving or suspending bendiocarb in a liquid and mixing the resultant solution or suspension with the absorbent base granules such that the solution or suspension is absorbed by the base granules to distribute the bendiocarb within the lattice structure of the base granules.

The base granules in the impregnated granules usually have a bulk density less than 1g per ml. The base granules preferably have a free-flow sorptive capacity for the liquid of at least 15 ml per 100g of the base granules.

The impregnated granules are preferably prepared by admixing the base granules with less than their free flow sorptive capacity of the solution or suspension of bendiocarb. When a suspension of bendiocarb is employed, this is conveniently obtained by admixing with water a wettable powder of bendiocarb, comprising bendiocarb admixed with surface active agents and optionally a particulate carrier. The wettable powder can contain for instance 75-80% bendiocarb. A sticker may also be

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admixed with the water. The sticker may be a water-soluble sticker. Examples of the stickers are gum acacia, cellulose ethers (e.g. sodium carboxymethyl cellulose), alginates, starches, ligninsulfonates, polyvinyl alcohol, polyvinyl acetate, sugars (e.g. dextrin or corn syrup), whey, or milk, preferably gum acacia, polyvinyl alcohol or polyvinyl acetate. The granules may contain 0.01-1% of the sticker. The sticker may consist of water-soluble sticker, though other sticker materials may also be present, e.g. in a content up to 25% by weight of total sticker.

The impregnated granules are preferably prepared by admixing the base granules with a solution of bendiocarb. The solubility of bendiocarb in the liquid employed is generally at least 150g per litre as measured at 15°C. The solution may be in a low volatile liquid, e.g. cyclohexanone. Preferably, however, the solution is in a high volatile liquid, e.g. dichloromethane, acetone or chloroform, especially dichloromethane.

After admixing the base granules with the solution or suspension of bendiocarb in a liquid, the liquid is generally either removed when it is high volatile or allowed to remain when it is low volatile. The use of a high volatile liquid which is removed, over a low volatile liquid which is not, is preferred to avoid slow

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evaporation of the low volatile liquid from the granules. The high volatile liquid should be sufficiently volatile so that it can be evaporated off conveniently from the granules which have absorbed the solution or suspension, e.g. by being placed in a forced air draught and periodically agitated or in a fluidised bed type drier and warm air passed through.

The impregnated granules are preferably prepared by dissolving bendiocarb in a high volatile liquid (e.g. dichloromethane to give a solution containing 200-220g, for instance 200g, of bendiocarb per litre), adding the resultant solution to corn cob grit revolving in a drum, and when the corn cob grit has absorbed the solution, evaporating off the liquid.

The impregnation with bendiocarb is usually carried out at ambient temperature, e.g. 15 to 20°C. A bendiocarb content up to 4% by weight in the granules can usually be obtained in a single stage impregnation. Higher contents usually require a multi-stage impregnation (i.e. repetition of the impregnation stage).

The impregnated granules may contain a slow release agent, so that in use the bendiocarb is released over an extended period of time. The slow release agent is preferably impregnated in the granules. Alternatively, however, the agent may coat the granules. The agent may

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be incorporated by impregnation and coating.

The slow release agent should be a low volatile, viscous and water-insoluble material, to enable it to perform its function in use. The agent usually has a viscosity of 10-4000, preferably 10-500, centipoises. Viscosities in this specification are as measured at 20°C on a Brookfield viscometer, RVT model, number 1 spindle, at 0.5 revolution per minute, unless otherwise stated.

The slow release agent is preferably a polymer. The agent may be for example an ester, e.g. dibutyl phthalate, or a hydrocarbon. The hydrocarbon and the viscosity of the agent, may be as discussed below in relation to a hydrocarbon sticker. When the slow release agent is a polymer, the polymer may be formed in the granule itself by applying monomer to the base granules and then polymerising the monomer. Usually, however, the agent is pre-formed. The agent may be applied in the same way that the bendiocarb may be applied. Indeed the agent may be applied together with the bendiocarb. Alternatively, the agent may be applied after the bendiocarb.

Where a slow release agent is employed in the impregnated granules, it usually represents 0.1 to 20% of the granules.

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The novel bendiocarb granules are preferably, however, not impregnated granules but coated granules, i.e. base granules to which the bendiocarb adheres by means of a sticker. Generally the coated granules contain 0.2-5% of sticker. Usually, the base granules have thereon a layer comprising the sticker and on the sticker layer is a layer comprising the bendiocarb. Usually, the coated granules are prepared by coating the base granules successively with a layer comprising the sticker and then with a layer comprising the bendiocarb.

The sticker may be for example a liquid polysaccharide, e.g. corn syrup or molasses, vegetable oil, e.g. corn oil or olive oil, or rosin.

Preferably the sticker comprises a hydrocarbon of viscosity at least 20 centipoises. The hydrocarbon is generally of viscosity 4000, preferably 20-500, centipoises. The hydrocarbon may be naturally occurring or may have been obtained synthetically, e.g. by polymerising one or more unsaturated monomers. Normal hydrocarbons may be used, though branched hydrocarbons are preferred. Aliphatic hydrocarbons are preferred. Mixtures of hydrocarbons may be employed. Preferably the sticker consists of the hydrocarbon, though other sticker materials may also be present e.g. in a content up to 25% by weight of the sticker.

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The hydrocarbon may be, for example, a mineral oil or 300 second fuel oil. Preferably, however, the hydrocarbon is a polymer of butene, which may be 1-butene, 2-butene or isobutene. Copolymers of 2 or of all 3 of these may be employed. Other comonomers may also be employed, though this is not preferred. Mixtures of the polymers may be used. Preferably the polymer of butene consists of polymerised butene, especially derived from a mixture of 1 and 2-butenes or from isobutene, and particularly preferred is the polymer consisting of polyisobutene. Commercial polyisobutenes, while consisting essentially of polymerised isobutene, often contain minor amounts, e.g. up to 10% by weight, of polymerised normal butene. For the present purpose, they are regarded as polyisobutene. The polymerised butene is conveniently a low viscosity liquid rather than a solid at ambient temperature. The polymerised butene is preferably of viscosity 20-500 cps, e.g. Hyvis* 05 (from BP Chemicals) which has a viscosity of 200 cps, Hyvis 04 (from BP Chemicals) which has a viscosity of 50 cps, or Indopol* L14 (from Amoco) which has a viscosity of 50 cps.

The choice of viscosity depends on the stickiness required and the ease and method of application of the hydrocarbon to the base granules. So that a layer of the hydrocarbon can be applied conveniently to base granules

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without using a diluent to reduce its viscosity, its viscosity is generally 20 to 500, preferably 40 to 150, centipoises, as measured at 20°C. Hydrocarbons of higher viscosity can be applied as solutions in an aliphatic or aromatic hydrocarbon solvent, e.g. kerosene, so that the viscosity of the solution is within this general range, but this is generally less convenient since for example slow evaporation of the solvent from the resultant granules can occur in storage. The viscosity of the hydrocarbon is preferably 20-500 cps at the temperature at which the hydrocarbon is applied to the base granules. The hydrocarbon can be applied to the base granules as an oil in water emulsion, with subsequent removal of the water by evaporation, though this technique is not preferred.

In the production of the coated granules, the bendiocarb is normally applied in powder form. It may be applied in admixture with a mineral filler such as China clay, precipitated silica or calcined Diatomite, and optionally a surface active agent, but this is not preferred. The bendiocarb is preferably hammer milled bendiocarb e.g. of particle size less than 50 microns.

The coated granules are preferably prepared by charging in turn to a flighted drum mixer, allowing time between additions, (i) the base granules, (ii) the

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sticker, and (iii) the bendiocarb.

The production of the coated granules is usually carried out at ambient temperature, though when a hydrocarbon of viscosity at least 20 centipoises is employed as sticker, the sticker and base granules may be preheated, e.g. to 35-40°C, prior to mixing to achieve a more even coating.

In a particular embodiment, the coated granules contain 1-15% bendiocarb and are corn cob grit granules having thereon a layer of polymerised butene of viscosity 20-300 cps, and on that layer a layer of bendiocarb.

The present novel granules are especially useful for application in admixture with brassica seed as discussed above. They are also surprisingly stable to chemical decomposition of the bendiocarb and resist agglomeration on storage.

The invention is illustrated by the following Examples.

Examples 1 and 2

Rape (Brassica napus, variety Tower) was sown in plots, 4.6 x 12.2 metres, arranged in a randomised block design replicated 9 times. The rape seed was premixed with granules containing 10% bendiocarb and was sown through the drill box of a commercial disc drill. The granules were impregnated granules consisting of 10%

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B bendiocarb and 90% Agsorb 24/48 S-100, and were of bulk density 0.99 g/ml and mean major dimension 450 microns, and 97% of the granules were of size 300-1000 microns in major dimension. The rape seed was sown at 5.6 kg per hectare and the bendiocarb applied at 140g (Example 1) or 280g (Example 2) of the active ingredient per hectare.

To assess the efficacy of the treatment in Example 2, 10 field collected flea beetles (Phyllotreta cruciferae) were placed in each of 6 cages over plants in each of 2 of the replicates, 10 (first exposure), 17 (second exposure) and 23 (third exposure) days after sowing, and the number of dead beetles recorded 24 and 48 hours after exposure. The cages were plastic cylinders 10 x 23cm with a nylon screen covering the top and two 5cm side vents. Quartz sand was added to each cage to a depth of 1cm to cover the soil in the bottom of the cage, in order to facilitate observations. The results, together with those from controls not treated with pesticide, are shown below.

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Bendiocarb Rate, g per ha	Percent Flea Beetle Mortality					
	First Exposure		Second Exposure		Third Exposure	
	24 hrs	48 hrs	24 hrs	48 hrs	24 hrs	48 hrs
280	99.2	100.0	80.8	90.8	25.2	47.3
0, Control	0.2	0.2	0.0	6.7	0.0	0.0

Seedling damage by flea beetles was assessed 4 weeks after sowing, on treated plots and on control plots not treated with pesticide, on a scale on which:

0 represents no damage;

0.5 represents 1-10% of leaf surface area damaged;

1 represents 11-25% of leaf surface area damaged;

2 represents 26-50% of leaf surface area damaged;

3 represents 51-75% of leaf surface area damaged;

4 represents 76-100% of leaf surface area damaged;

The results are shown below.

Example	Bendiocarb Rate, g per ha	Seedling Damage Rating
1	140	0.04
2	280	0.15
Control	0	2.48

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Example 3

A 25kg batch was prepared, consisting of:

Bendiocarb	:	5%	
Indopol L-14	:	3%	(Amoco)
Grit-O'Cobs* 101H	:	92%	(Corn cob grit by The Andersons)

The corn cob grit was charged to a double-cone shaped mixer and the Indopol L-14 sprayed on over 1 minute, and mixed for 5 minutes. The bendiocarb was added in 3 equal batches, mixing for 2 minutes between each addition, and finally for 10 minutes.

The resulting granules were of bulk density 0.52 g/ml and mean major dimension 1500 microns, and 98% was of size in the range 1000-2000 microns.

Example 4

A 50kg batch was prepared, consisting of:

Bendiocarb	:	5%	
Corb cob 12/20	:	95%	(from Mount Pulaski)

The corn cob grit was charged to a double-cone shaped mixer. The bendiocarb was dissolved in dichloromethane to give a 140g per litre solution. One half of this solution was sprayed onto the corn cob grit over 10 minutes and allowed ~1½ hours to mix and permit removal of the bulk of the solvent. The second half of the bendiocarb solution was then added and allowed

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1-1½ hours to mix. The solvent was allowed to evaporate at ambient temperature.

The resulting granules were of bulk density 0.52 g/ml and of mean major dimension 1000 microns, and 98% was of size in the range 600-1400 microns.

Examples 5-7

The granules prepared in Example 4 were tested in a similar way to Examples 1 and 2.

The results are shown below:

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Bendiocarb Rate, g per ha	Percent Flea Beetle Mortality					
	First Exposure		Second Exposure		Third Exposure	
	24 hrs	48 hrs	24 hrs	48 hrs 72 hrs	24hrs	48 hrs 72 hrs
140	50.9	89.9	28.2	28.2 48.2	23.1	42.9 42.9
280	57.4	72.1	59.4	59.4 72.1	34.8	69.2 71.2
0,Control	0.0	0.0	0.0	1.0 12.0	0.0	3.2 5.8

Example	Bendiocarb Rate, g per ha	Seedling Damage Rating
5	71	1.5
6	140	0.6
7	280	0.3
Control	0	4.0

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THE EMBODIMENTS OF THE INVENTION IN WHICH AN EXCLUSIVE PROPERTY OR PRIVILEGE IS CLAIMED ARE DEFINED AS FOLLOWS:

1. A method of combating pests at locus at which a brassica crop is growing or is to grow, comprising applying to the locus a pest-combating amount of granules containing bendiocarb wherein,

a) no more than 4% by weight of the granules is smaller than 250 microns in major dimension,

b) the granules contain 1 to 15% by weight of bendiocarb,

c) the granules have bulk density of 0.4 to 0.8 g/ml as measured according to CIPAC test method MT 58.4, and

d) the granules have a mean major dimension of 1000 to 2000 microns.

2. A method according to claim 1 wherein 50-1000g of bendiocarb are applied per hectare.

3. A method according to claim 1 wherein the brassica crop is rape.

4. A method according to claim 1 wherein a mixture of the granules and the seed of the brassica crop is sown together.

5. A method according to claim 4 wherein the proportion of granules:seed is from 1:10 to 10:1 by weight.

6. A method according to claim 4 wherein the granules are of similar size and bulk density to the brassica seed.

7. A method according to claim 4 wherein the granules and seed are each of bulk density 0.4-0.8g/ml and of mean major dimension 1000-2000 microns.

8. A method according to claim 7 wherein the bulk density is 0.45-0.60g/ml.

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9. A method according to claim 8 wherein the seed is rape seed.

10. Granules containing bendiocarb, wherein,

a) no more than 4% by weight of the granules is smaller than 250 microns in major dimension,

b) the granules contain 1 to 15% by weight of bendiocarb,

c) the granules have bulk density of 0.4 to 0.8 g/ml as measured according to CIPAC test method MT 58.4, and

d) the granules have a mean major dimension of 1000 to 2000 microns.

11. Granules according to claim10 whose bulk density is 0.45-0.60 g/ml.

12. Granules according to claim 10 which are botanical base granules impregnated with the bendiocarb, or botanical base granules to which the bendiocarb adheres by means of a sticker.

13. Granules according to claim 12 wherein the botanical base granules are corn cob grit.

14. Granules according to claim 10 which are base granules to which the bendiocarb adheres by means of a sticker.

15. Granules according to claim 14 wherein the base granules have thereon a layer comprising the sticker and on the sticker layer is a layer comprising the bendiocarb.

16. Granules according to claim 14 or 15 wherein the granules contain 0.2-5% by weight of the sticker.

17. Granules according to claim 14 or 15 wherein the sticker comprises a hydrocarbon of viscosity 20-4000 centipoises.

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18. Granules according to claim 14 or 15 wherein the sticker comprises a polymer of butene, which polymer has a viscosity of 20-4000 centipoises.

19. Granules according to claim 14 or 15 wherein the sticker comprises polyisobutene of viscosity 20-4000 centipoises.

20. A method of preparing granules impregnated with bendiocarb, which granules are of bulk density 0.4-0.8g/ml and of mean major dimension 1000-2000 microns, which method comprises dissolving or suspending bendiocarb in a liquid and mixing the resultant solution or suspension with absorbent base granules such that the solution or suspension is absorbed by the base granules to distribute the bendiocarb within the lattice structure of the base granules.

21. A method of preparing granules containing bendiocarb, which granules are of bulk density 0.4-0.8g/ml and of mean major dimension 1000-2000 microns, the granules being base granules having thereon a layer comprising the sticker and on the sticker layer a layer comprising bendiocarb, which method comprises coating the base granules successively with a layer comprising the sticker and then with a layer comprising bendiocarb.

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